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HIGH TEMPERATURE COMPOSITES USING SCHIFF'S BASE PYROPOLYMER MATRICES

by

Steven J. Thoman and Leonard J. Buckley

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1.0 SUMMARY

Two acetylene functional Schiff's base pyropolymers have been evaluated as matrix materials for fiber reinforced composite materials. Autoclave processing techniques were developed to produce high quality laminated composites. These matrix resins can be made conductive through post-cure heat treatments and were studied in a conductivity range from 10^{-10} to 10^0 S/cm. The insulating resin composites have very stable mechanical properties up to 300°C ; however, these properties are significantly lower than state-of-the-art polyimides. The post-processing heat treatments required to make the pyropolymers conductive caused extensive transverse matrix cracking in the laminates.

2.0 INTRODUCTION

Organic composite materials are currently being used in both flight critical and secondary applications on Navy aircraft. As aircraft technology has advanced, there has been a demand for structural composites that are capable of operating in higher temperature regimes. Applications of epoxy matrix composite structures are limited to 140°C , bismaleimide structures to 180°C , and polyimide structures to 300°C . These materials have been extensively characterized and have been used in appropriate aircraft components [1-4].

Acetylene functional resins offer the potential for enhanced high temperature composite applications. Various monomers have been synthesized and polymerization processes for many of these monomers have been explored [5-9]. Walton has shown that acetylene functional Schiff's base resins can be heat treated after polymerization to impart environmentally stable electrical conductivity [7]. The conductivity level was increased from an insulator (10^{-10}

S/cm) to a conductor (10^0 S/cm) by post curing the resin. Rossi, et al., have recently reported on the thermal oxidative stability of these resins [8,9]. This study includes process development and assessment of the mechanical performance of two graphite reinforced acetylene functional Schiff's base resins. The use of these Schiff's base acetylene functional pyropolymers as composite matrix resins has been examined at a wide range of conductivities.

3.0 EXPERIMENTAL APPROACH

The acetylene terminated Schiff's base monomers were supplied in powder form by National Starch and Chemical Corporation, Bridgewater, NJ and two isomers were investigated (Figures 1 and 2). Thermcon 1000 (T1000) and Thermcon 2000 (T2000) are designated N,N'-[1,4-phenylenedimethylidene bis-(3-ethynylaniline)] and N,N'-[1,3-phenylenedimethylidene bis-(3-ethynylaniline)], respectively. Cure cycles were not available for processing these resins as matrices in fiber reinforced composites; therefore, the initial portion of this study involved development of a processing schedule germane to composite materials. A test plan was developed to evaluate the mechanical performance and upper use temperature of the materials. Tension, interlaminar shear, and fracture toughness tests were performed. The composite fracture surfaces were examined with a scanning electron microscope to determine the mechanisms of failure. The thermal capabilities of the composite materials were investigated with Dynamic Mechanical Analysis (DMA), Thermal Gravimetric Analysis (TGA) and thermal cycling exposure.

3.1 COMPOSITE PROCESSING

Differential scanning calorimetry (DSC), autoclave processing, nondestructive evaluation, and optical microscopy were used to develop and

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verify the cure cycles for the composite materials. A DuPont 1090 thermal analysis system with a DSC module was used to monitor the heats of reaction of the two resins during cure. T1000 and T2000 monomers were heated from 25°C to 300°C at 1.5°C per minute and composite cure cycles were developed from the DSC results.

Composite laminates were prepared for cure by stacking layers of AS4 5-harness woven graphite cloth and powdered resin in approximately a 40% resin volume combination (Figure 3). The laminate was sandwiched between two layers of porous release film and then a no-bleed bagging arrangement was added (Figure 4).

Cured laminates were nondestructively inspected with pulse echo ultrasonics. The quality of the composites was also assessed with photomicroscopy of polished laminate cross-sections. Photomicrographs were taken with a Nikon Optophot-M optical microscope.

Some of the laminates received additional heat treatments to impart conductivities to the matrix resins. Post-cure heat treatments were performed at 500°C and 600°C for 100 hours and a nitrogen purge was used to prevent oxidative degradation of the polymers. Walton has shown that the conductivities of the unreinforced resins after 100 hour heat treatments at 500°C and 600°C are 10^{-5} S/cm and 10^0 S/cm, respectively [6].

3.2 THERMAL PROPERTY EVALUATION

The thermal performance of the reinforced T1000 and T2000 resins was evaluated by Dynamic Mechanical Analysis (DMA) and Thermal Gravimetric Analysis (TGA). The DMA was run at 5°C per minute from 25°C to 500°C. The TGA was run at 0.5°C per minute from room temperature to 500°C.

Microcracking susceptibility of the composite materials were assessed by examining the microstructure after thermal cycling. Composite specimens were cycled from room temperature to 300°C with a five minute soak and an average heat/cool rate of 60°C per minute.

3.3 MECHANICAL PROPERTY EVALUATION

The mechanical property evaluation of the composite laminates consisted of interlaminar shear, tension, and Mode I fracture toughness tests. Interlaminar shear was evaluated with the ASTM D2344-76 short beam method. Testing was performed at room temperature, 200°C, and 300°C conditions on AS4/T1000, AS4/T2000, and AS4 unsized/T1000 composite specimens. Tension tests were performed at room temperature on the AS4/T1000 material using the ASTM D3039-76 test method. Mode I fracture toughness of AS4/T1000 was evaluated with the double cantilever beam method [10].

4.0 DISCUSSION OF RESULTS

4.1 NON-CONDUCTIVE RESIN STATE

4.1.1 COMPOSITE PROCESSING

A DSC plot of T1000 resin heated at 1.5°C per minute is shown in Figure 5. The melt temperature was determined to be 138°C and the exotherm initiated at 148°C. A preliminary cure cycle was developed for T1000 composite materials and is shown in Figure 6. A short hold was included after the melting point to allow the temperature distribution to stabilize within the part before initiation of the cure reaction. The DSC plot in Figure 7 shows that little reaction occurred during this hold.

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Initial attempts to cure the AS4/T-1000 composites using the preliminary cure cycle resulted in laminates with high void contents (Figure 8). The large surface area inherent in powders caused the T-1000 resin to absorb moisture during laminate fabrication. Heating the resin under a dynamic vacuum oven at 65°C for 24 hours before laminate fabrication eliminated the absorbed moisture and produced laminates that contained low void contents. Subsequent composite laminates were processed with dried resins.

Microcracking occurred throughout the laminate's cross-section in the AS4/T1000 composites that were processed with the preliminary cure cycle (Figure 9). The cure cycle was altered to reduce the hold temperature from 220°C to 155°C and microcracking was significantly reduced (Figures 10 and 11). The lower cure temperature decreased the temperature at which gelation occurred and helped to reduce the thermal stresses that resulted during cooling to room temperature. Thermal analysis was performed to ensure that the material had fully reacted during this improved cure.

A DSC plot of T2000 resin heated at 1.5°C per minute is shown in Figure 12. The melt temperature was determined to be 85°C and the exotherm initiated at 158°C. AS4/T2000 laminates were processed in an autoclave using the cure schedule shown in Figure 13. Low void laminates were produced and no microcracking was apparent (Figure 14).

4.1.2 THERMAL PROPERTIES

DMA in air shows that the moduli of the T1000 and T2000 composites remain stable up to approximately 410°C (Figures 15 and 16). TGA shows composite weight loss in air initiates at approximately 340°C for both resins (Figures 17 and 18). Thermal cycling results indicate that microcracking had initiated after ten thermal cycles in both resin systems (Figures 19 and 20).

4.1.3 MECHANICAL PROPERTIES

The interlaminar shear stress of AS4/T1000 was determined to be 19.75 MPa at room temperature which is 100-300% lower than state of the art thermoset materials reinforced with a similar graphite weave [3]. The AS4/T2000 had interlaminar shear failure stresses that were similar to the values achieved by the AS4/T1000 composite (Table 1). Both materials exhibited stable interlaminar shear strengths up to 300°C. Interlaminar shear properties improved over room temperature values by 7.1% at 200°C and 18.7% at 300°C for AS4/T1000. Elimination of the sizing from the AS4 fibers caused little change in the ILS strengths obtained by the sized-fiber laminates. Tensile failure stress in the warp direction of the 5-harness AS4/T1000 was found to be 475MPa. Scanning electron microscopy of the tensile fracture surfaces (figure 21) show that fiber/matrix bonding was poor. The tensile specimens failed in a delamination mode before individual ply failure caused ultimate failure of the specimen (Figure 22). Double cantilever beam testing showed that the strain energy release rate of AS4/T1000 was 0.98 in-lb./in², significantly lower than state of the art organic composite materials reinforced with woven graphite [12]. Post failure fractography of the fracture surfaces showed that there was poor fiber/matrix adhesion (Figure 23). The failure tended to propagate along the fiber/matrix interface except at the small resin rich tow overlap areas where failure was forced into the matrix. Attempts were made to measure the fracture toughness of the unreinforced resin with compact tension specimens; however, notching the specimens caused a crack to propagate through the entire specimen. Therefore, the unreinforced pyropolymer is so brittle that its fracture toughness can not be measured with this test method.

4.2 CONDUCTIVE RESIN STATE

T1000 and T2000 laminates were prepared for ILS testing after exposure to the heat treatments required to render the materials conductive. These specimens could not be cut with a diamond grit saw blade on a milling machine without causing extensive delamination. Therefore, mechanical testing of these laminates was not performed. Samples for microscopy were cut with a cooled, rate controlled wafering machine to insure no damage was induced. Transverse microcracks were found in each of the laminates as shown in Figure 24.

5.0 CONCLUSIONS AND RECOMMENDATIONS

The processing techniques developed for graphite reinforced Thermcon resin matrix composites are less complex and less time consuming than state of the art polyimides [13]. A typical PMR-15 cure and post-cure are shown in Figure 25 for comparison. The initial mechanical properties generated for these material systems in the insulating state are significantly lower than those of state of the art polyimides [3]. The failures propagated through the fiber/matrix interfacial region indicating poor bonding characteristics. The development of a more suitable sizing will improve the fiber/matrix bonding, provide better translation of stresses, and result in improvements to the mechanical properties. Increasing the molecular weight between crosslinks through the use of chain extending oligomers may act to toughen this resin system, reduce its tendency to microcrack, and enable it to be used as a composite matrix. Post-curing the graphite reinforced pyropolymer laminates to produce conductive composites resulted in laminates that are not capable of being used in load-bearing structural applications.

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TABLE 1 - INTERLAMINAR SHEAR STRENGTHS (MPa)

	AS4/T1000 (sized)	AS4/T1000 (unsized)	AS4/T2000 (sized)	T300/PMR15
25°C	20.0	20.7	22.8	51.8
200°C	21.4	24.2	24.1	--
300°C	23.5	26.2	24.1	35.2

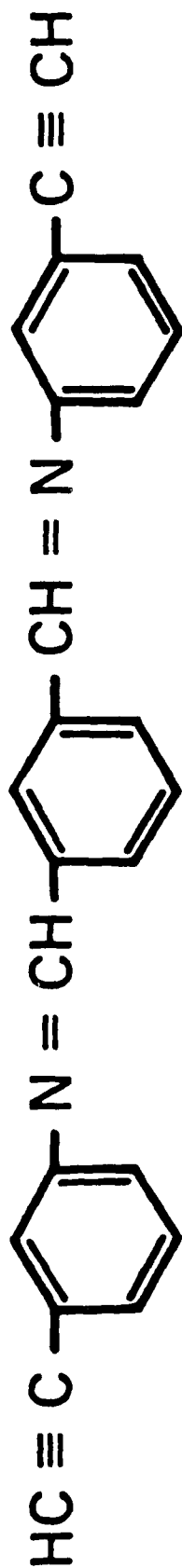


Figure 1. Thermcon 1000 Chemical Structure

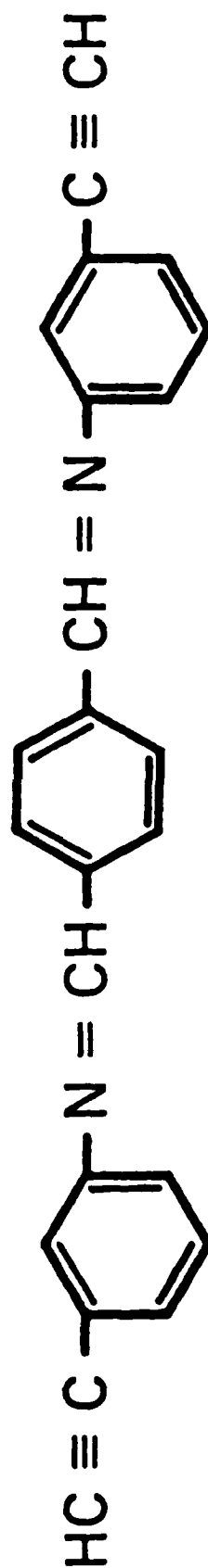


Figure 2. Thermcon 2000 Chemical Structure

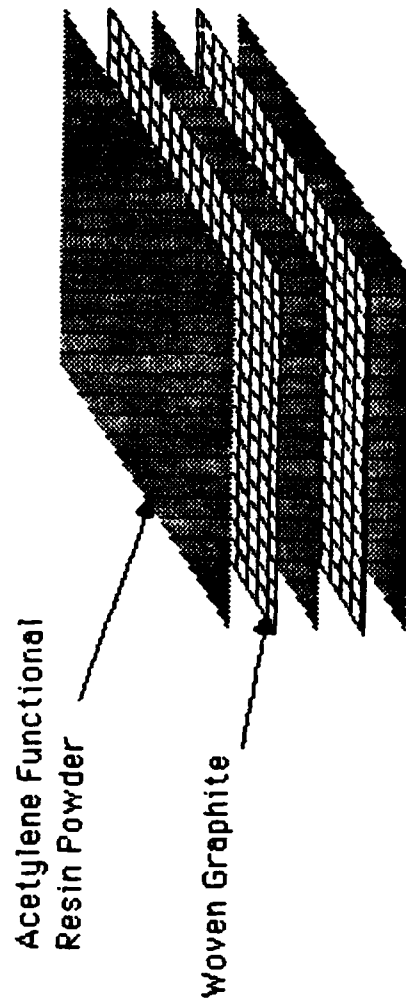


Figure 3. Composite Lamination Method

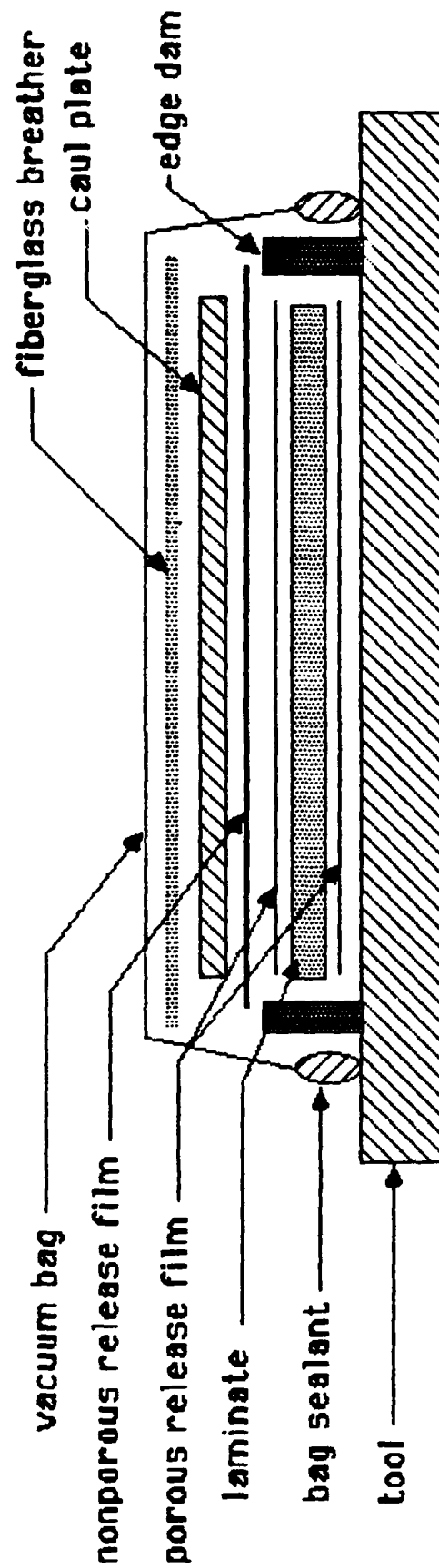


Figure 4. Bagging Arrangement

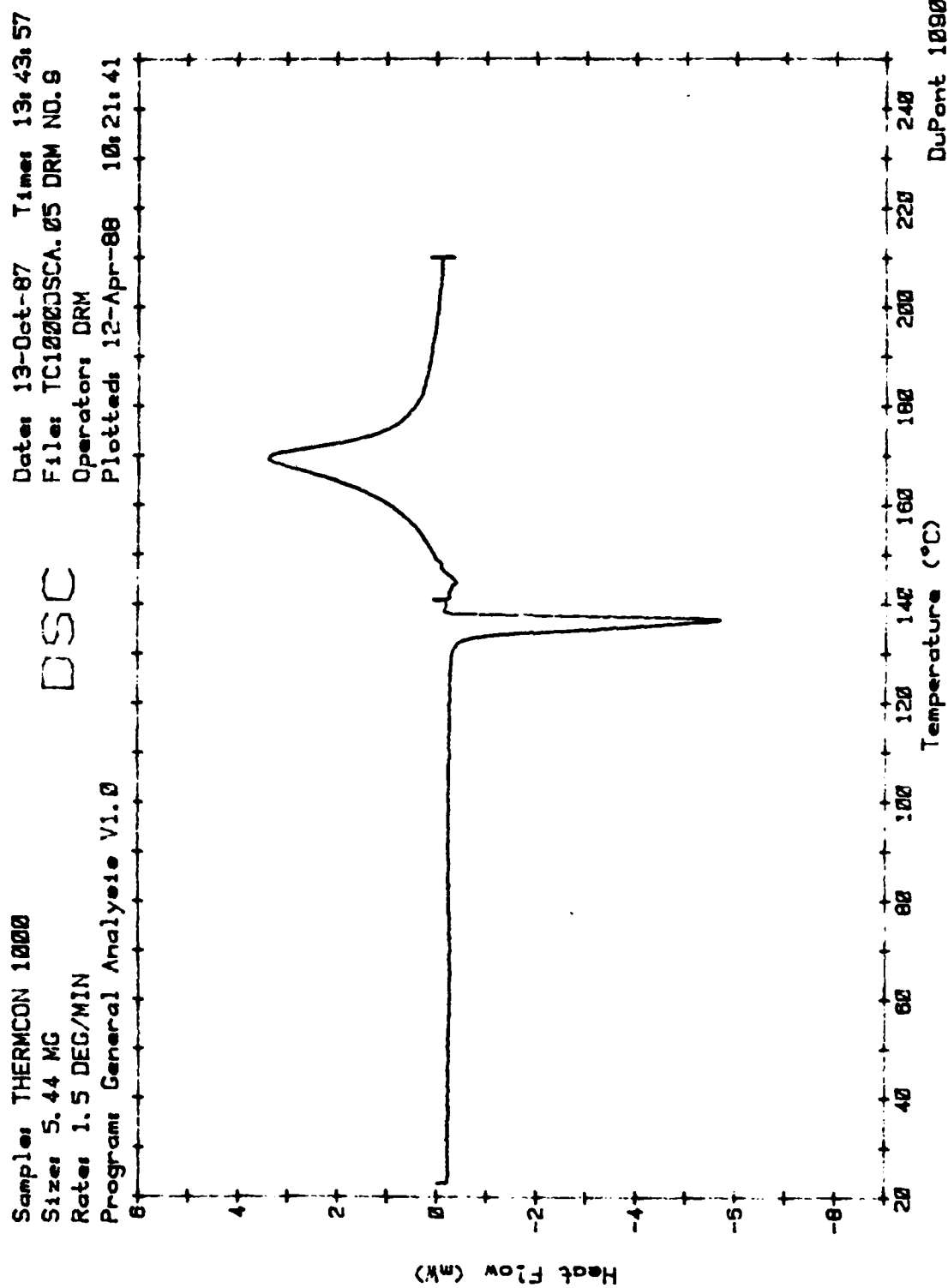


Figure 5. DSC Plot of T1000

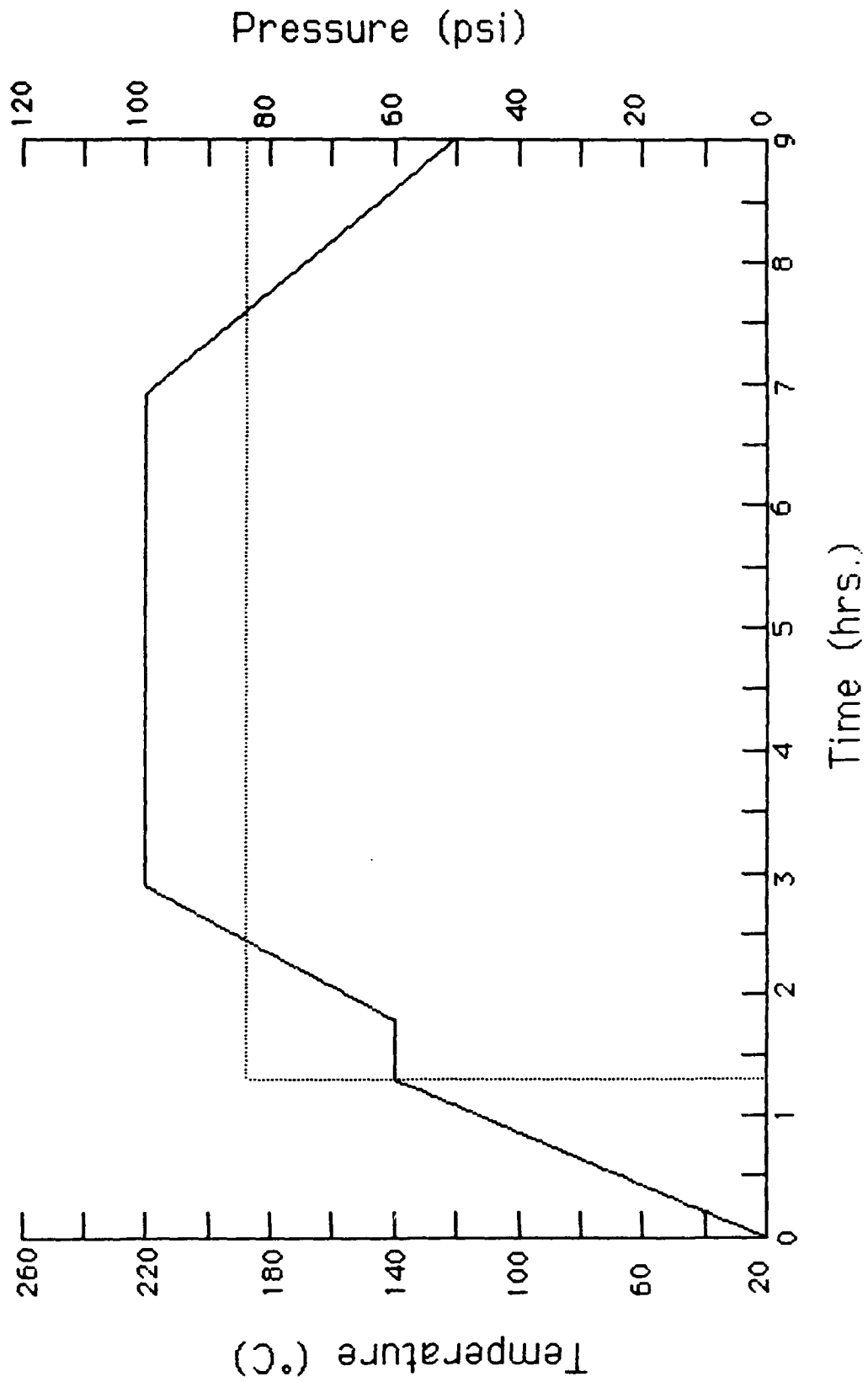


Figure 6. Preliminary T1000 Cure Cycle

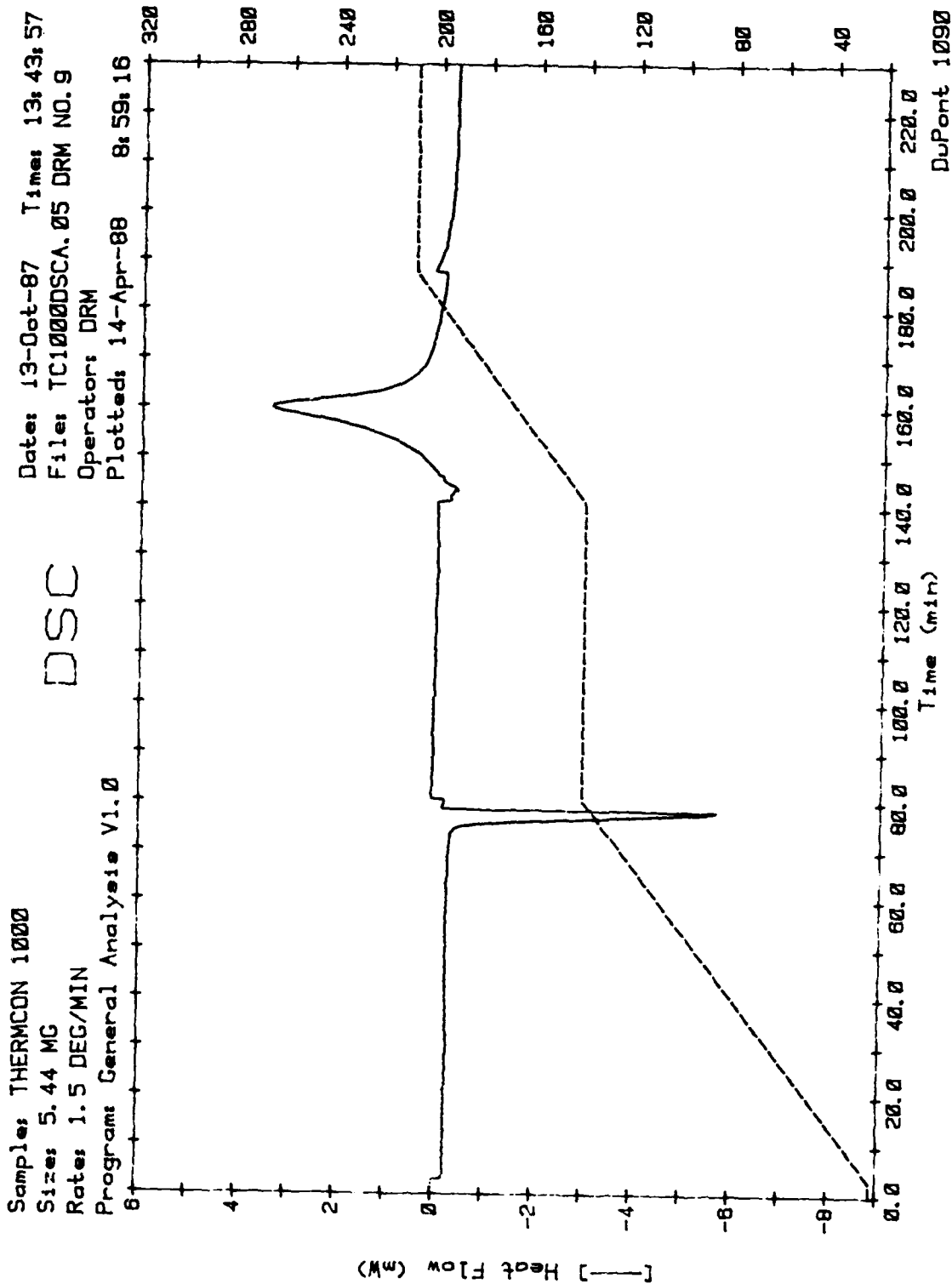


Figure 7. Effect of Post Melt Hold on Cure

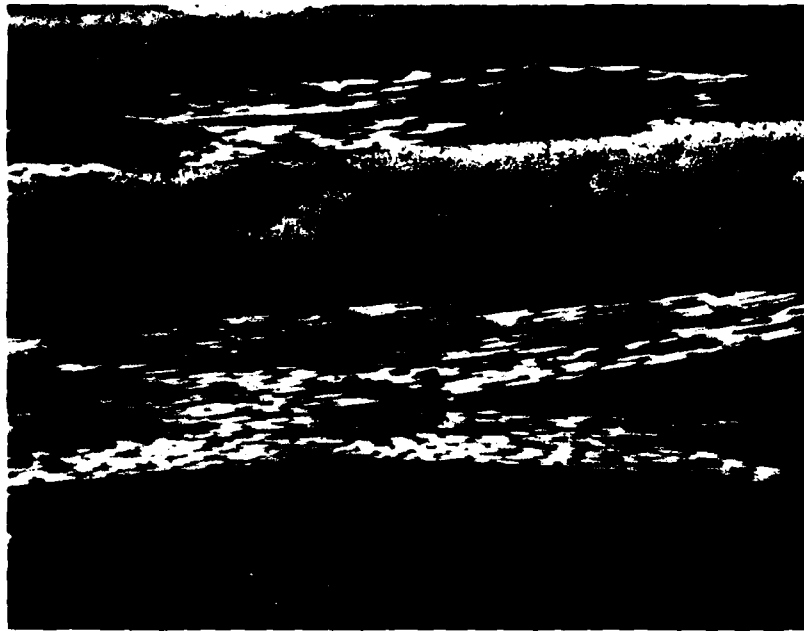


Figure 8. Voided AS4/T1000 Laminate (75x)

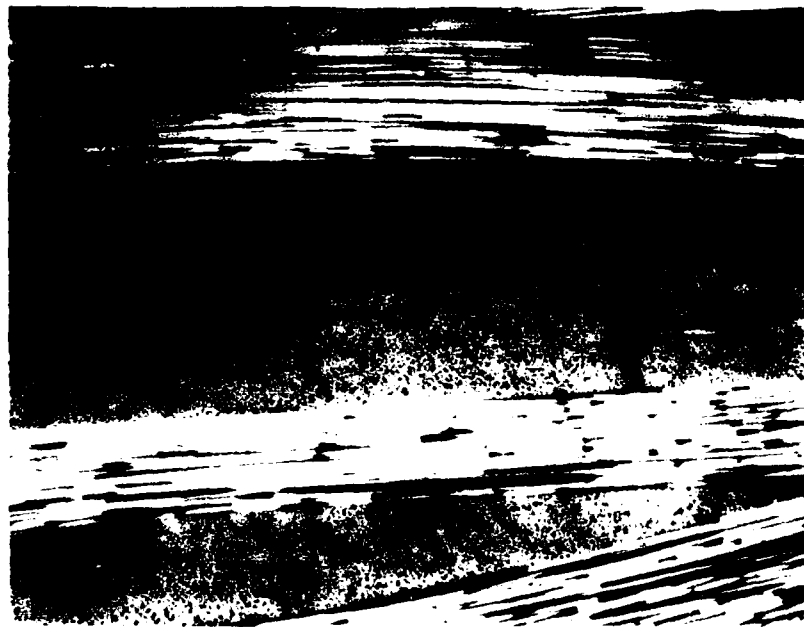


Figure 9. Microcracked AS4/T1000 Laminate (75x)

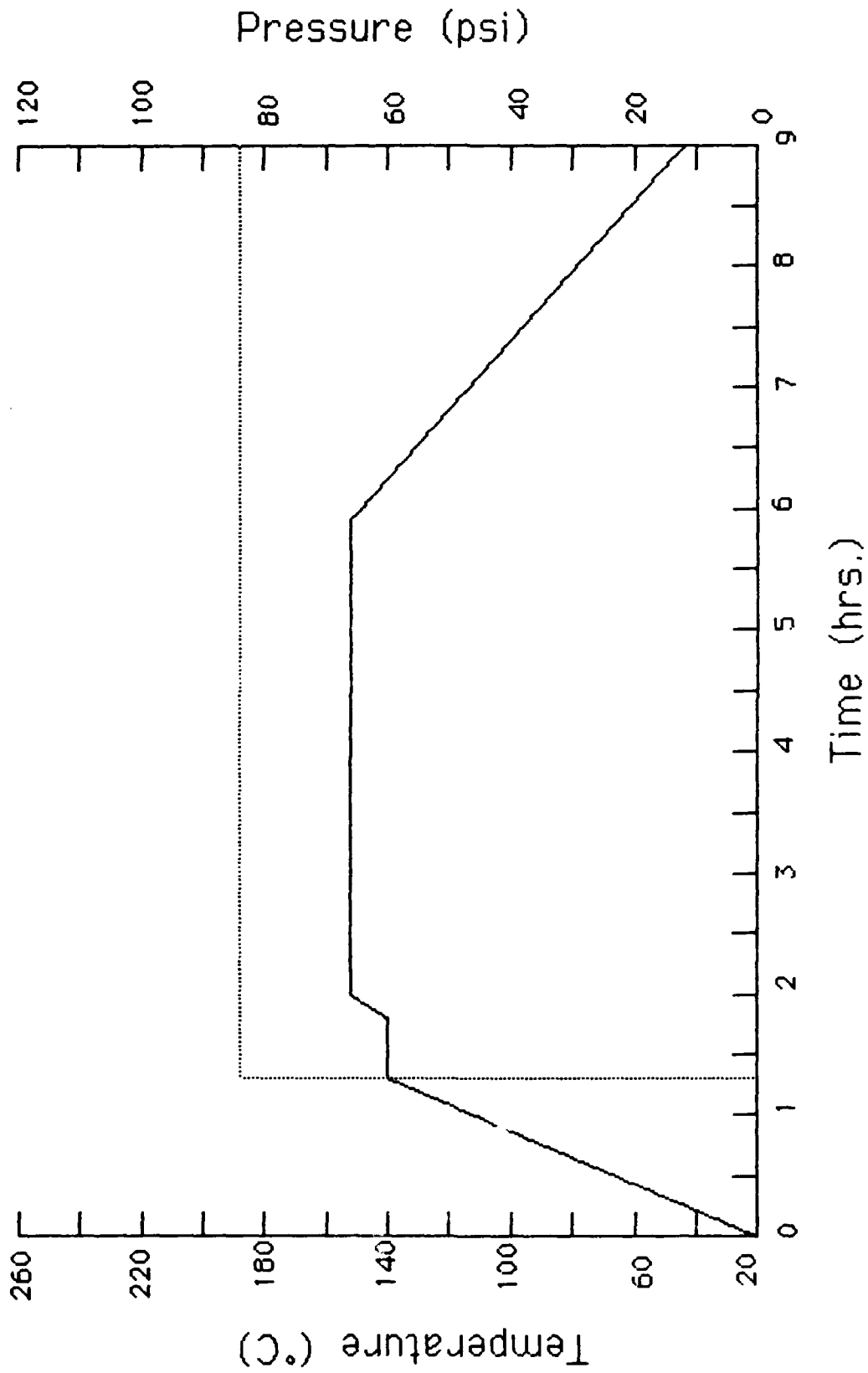


Figure 10. Improved T1000 Cure Cycle

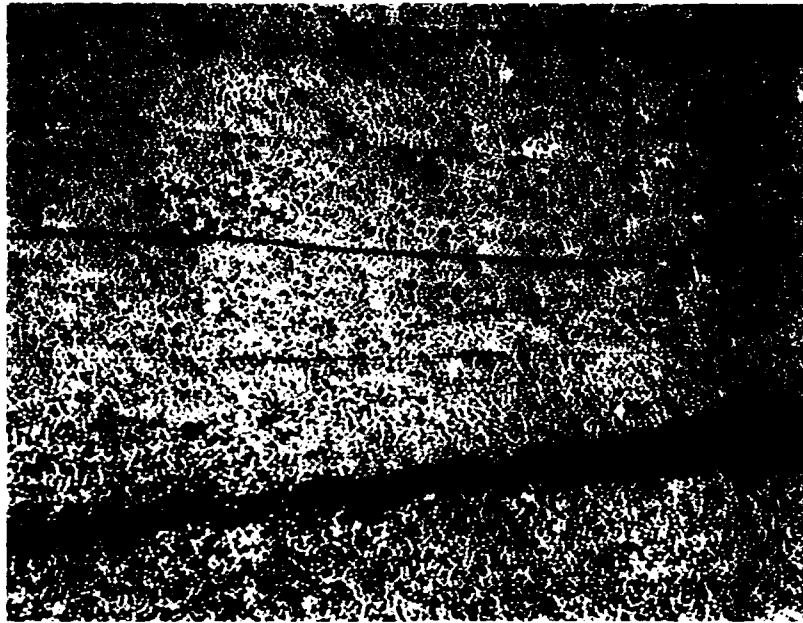


Figure 11. Improved AS4/T1000 Laminate (75x)

DSC

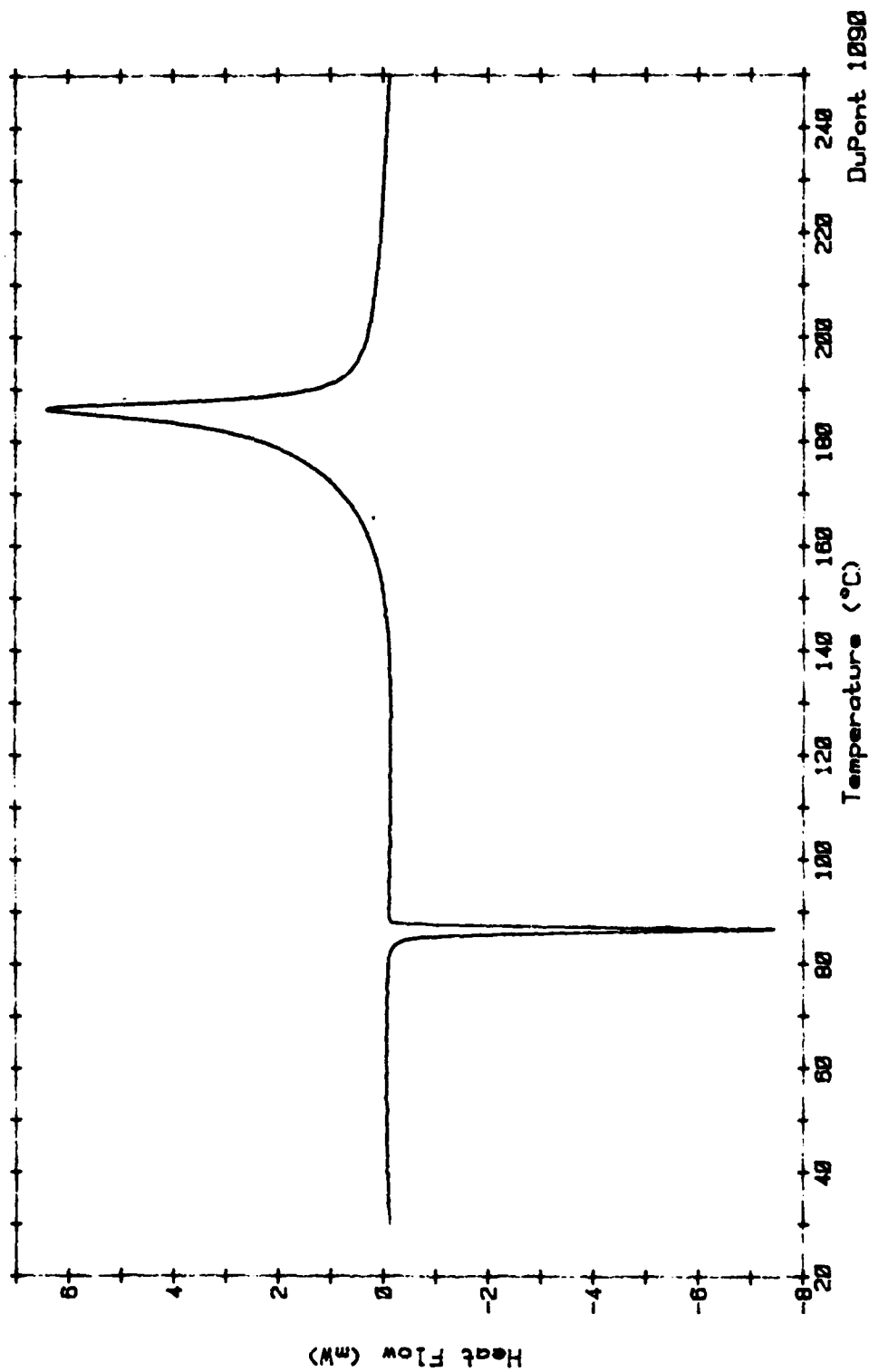


Figure 12. DSC Plot of T2000

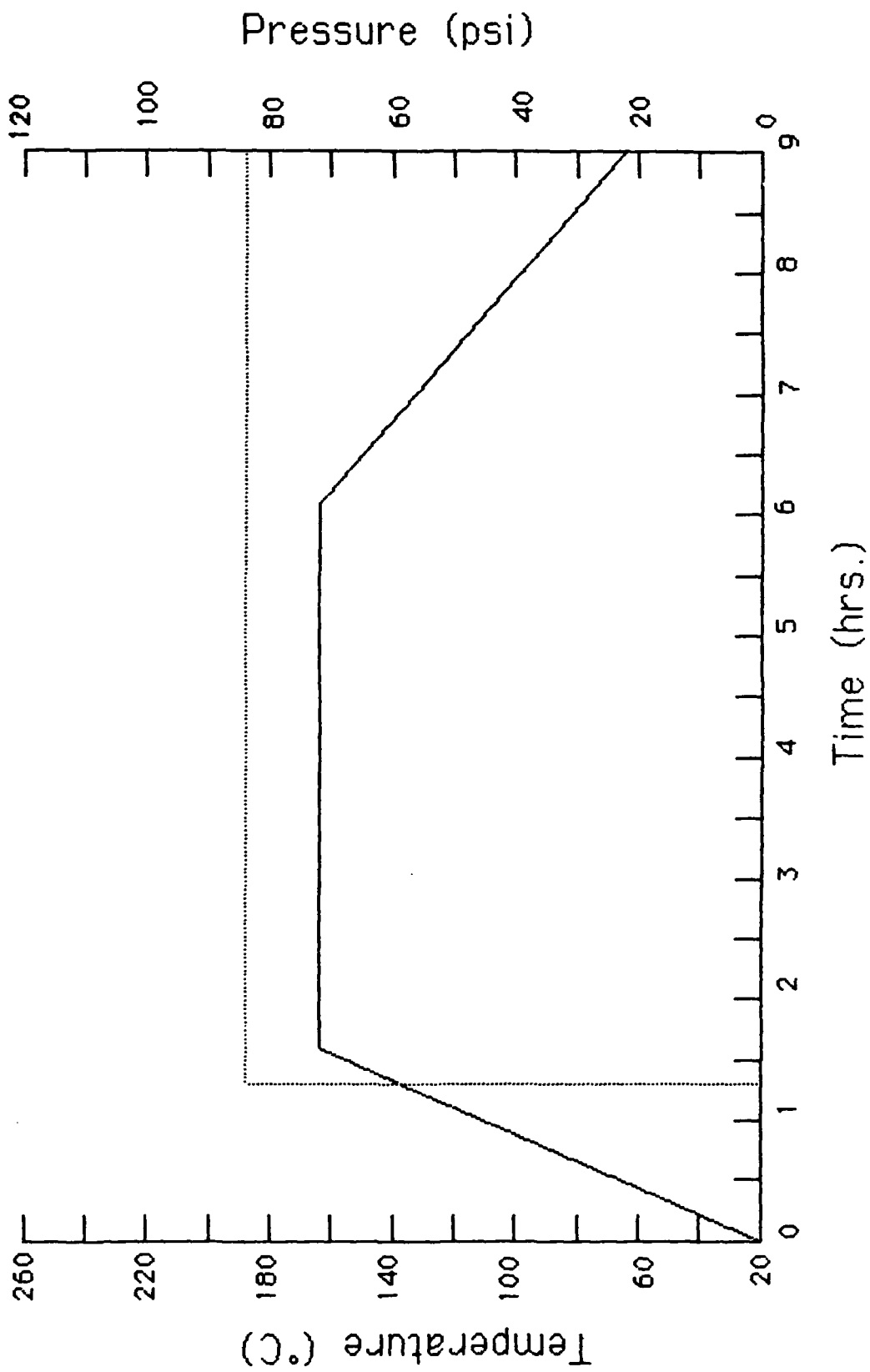


Figure 13. T2000 Composite Cure Cycle

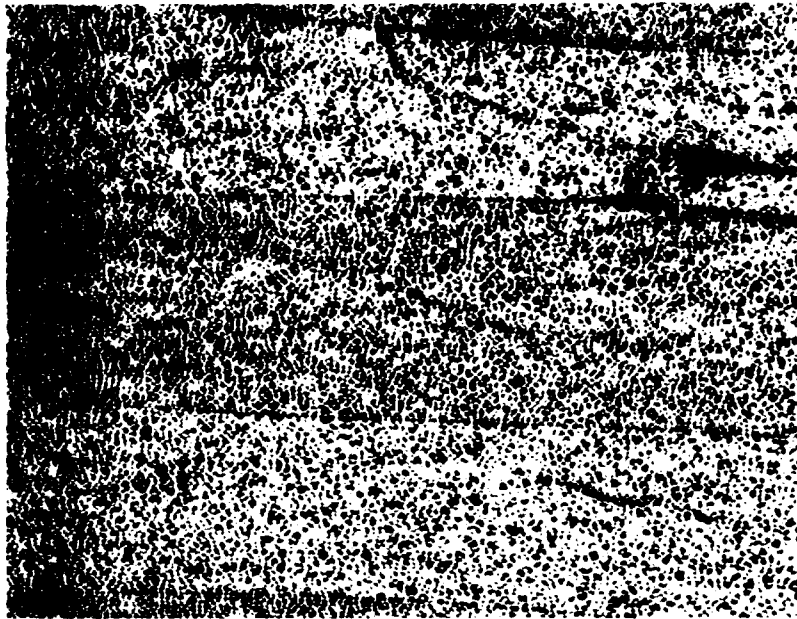


Figure 14. AS4/T2000 Laminate (75x)

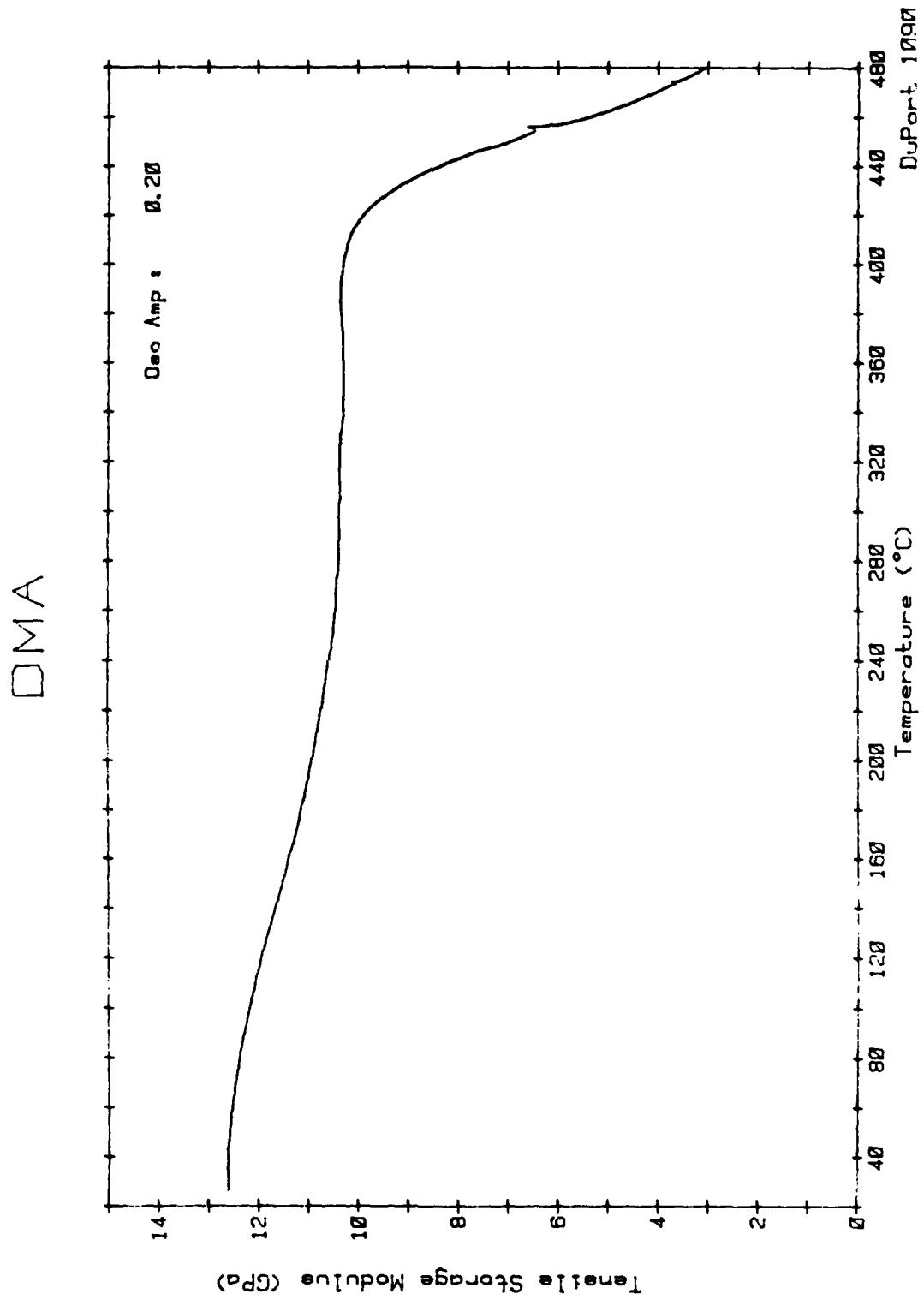


Figure 15. DMA in Air of AS4/T1000

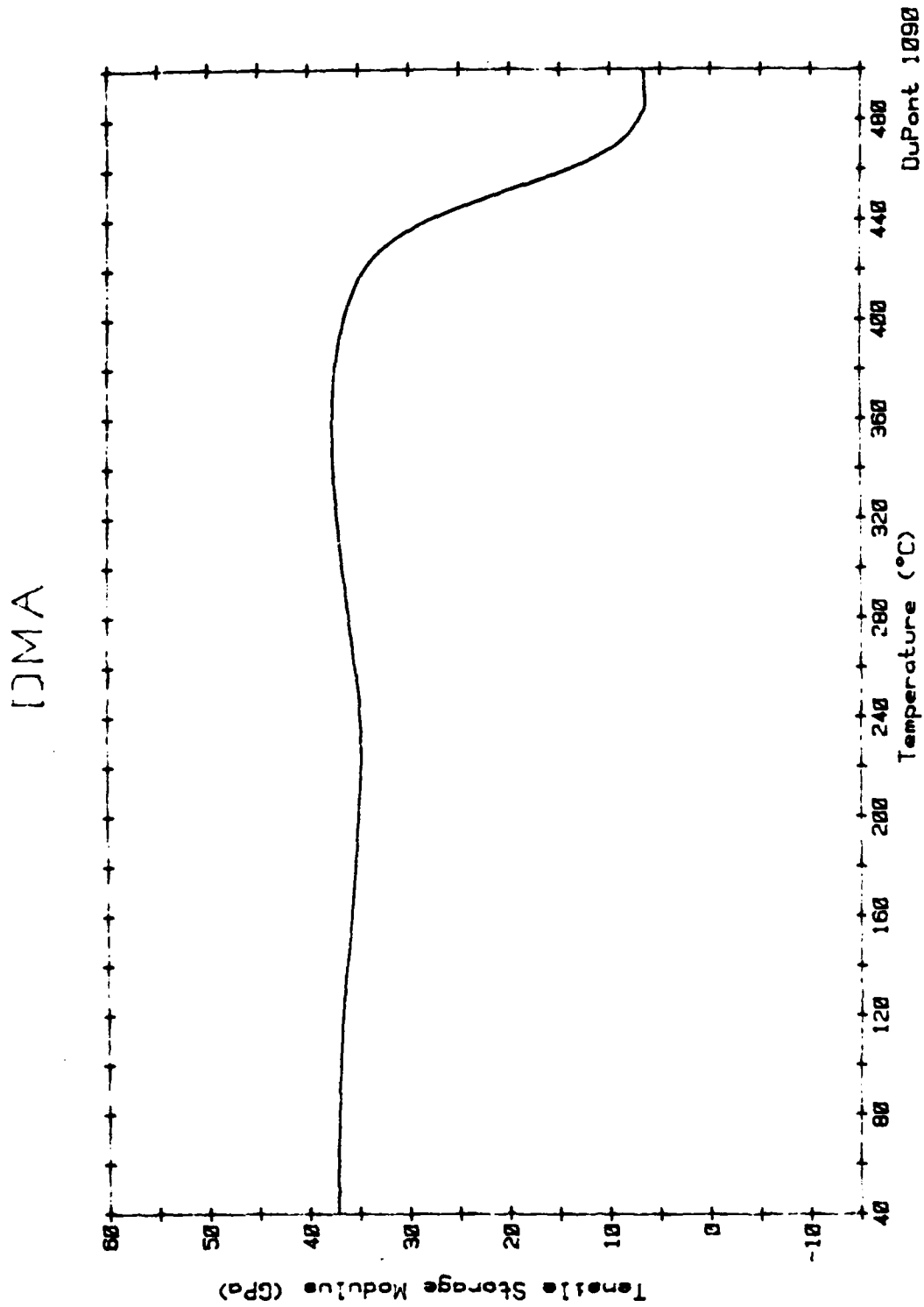


Figure 16. DMA in Air of AS4/T2000

Sample: AS4-SH/THERMCON 1000
 Size: 17.54 mg
 Rate: 0.5 DEG/MIN
 Program: TGA Analysis V2.0

TGA

Date: 21-Apr-88 Time: 16:51:14
 File: AS4/T1000.01 DSC 88-05
 Operator: DRM
 Plotted: 21-Apr-88 9:05:22

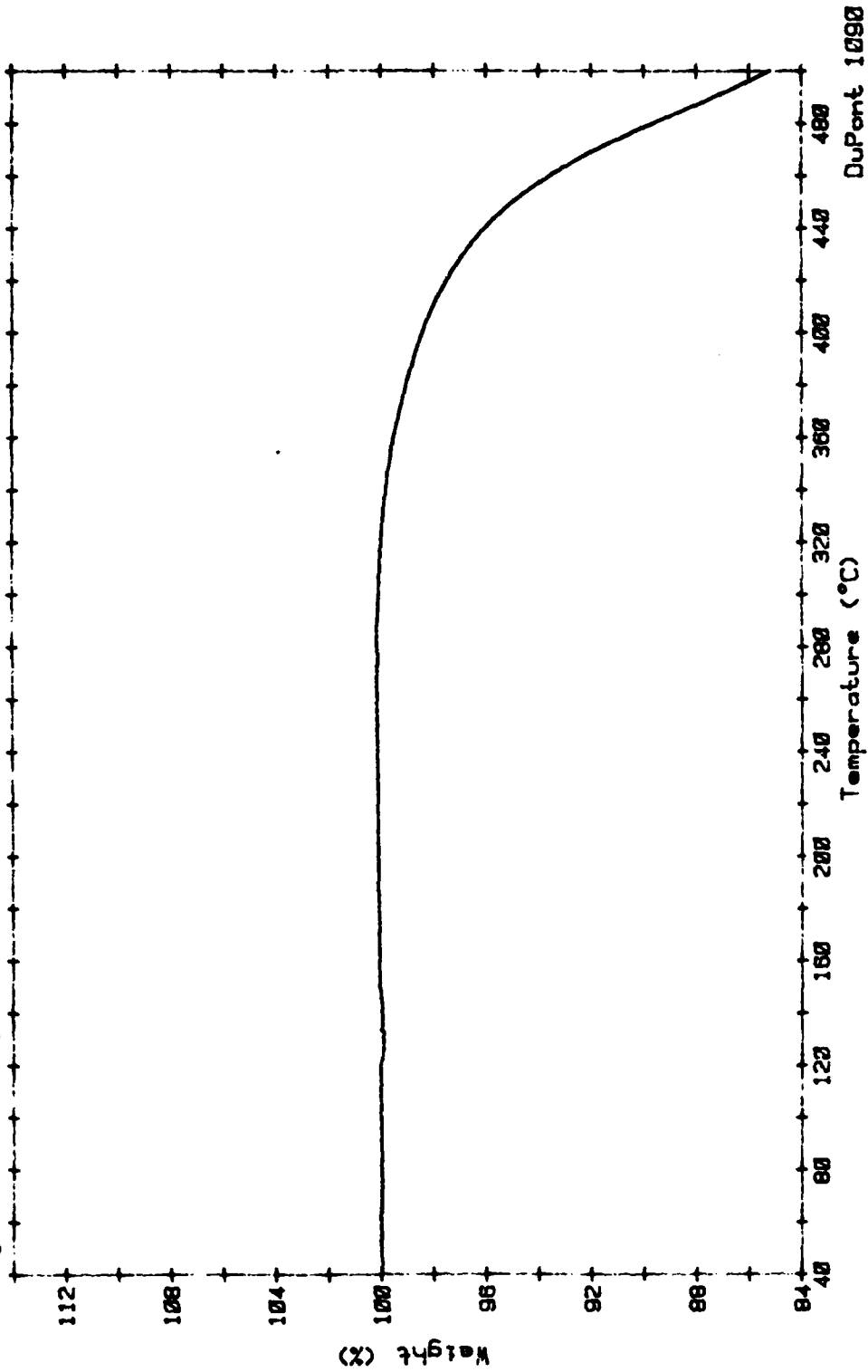


Figure 17. TGA in Air of AS4/T1000

Date: 12-Apr-88 Time: 12:10:45
 File: AS4-5HTGA.01 DSC 88-05
 Operator: DRM
 Plotted: 13-Apr-88 5:53:46

Sample: AS4-5H TC2000
 Size: 12.64 mg
 Rate: 0.5 DEG/MIN
 Program: TGA Analysis V2.0

TGA

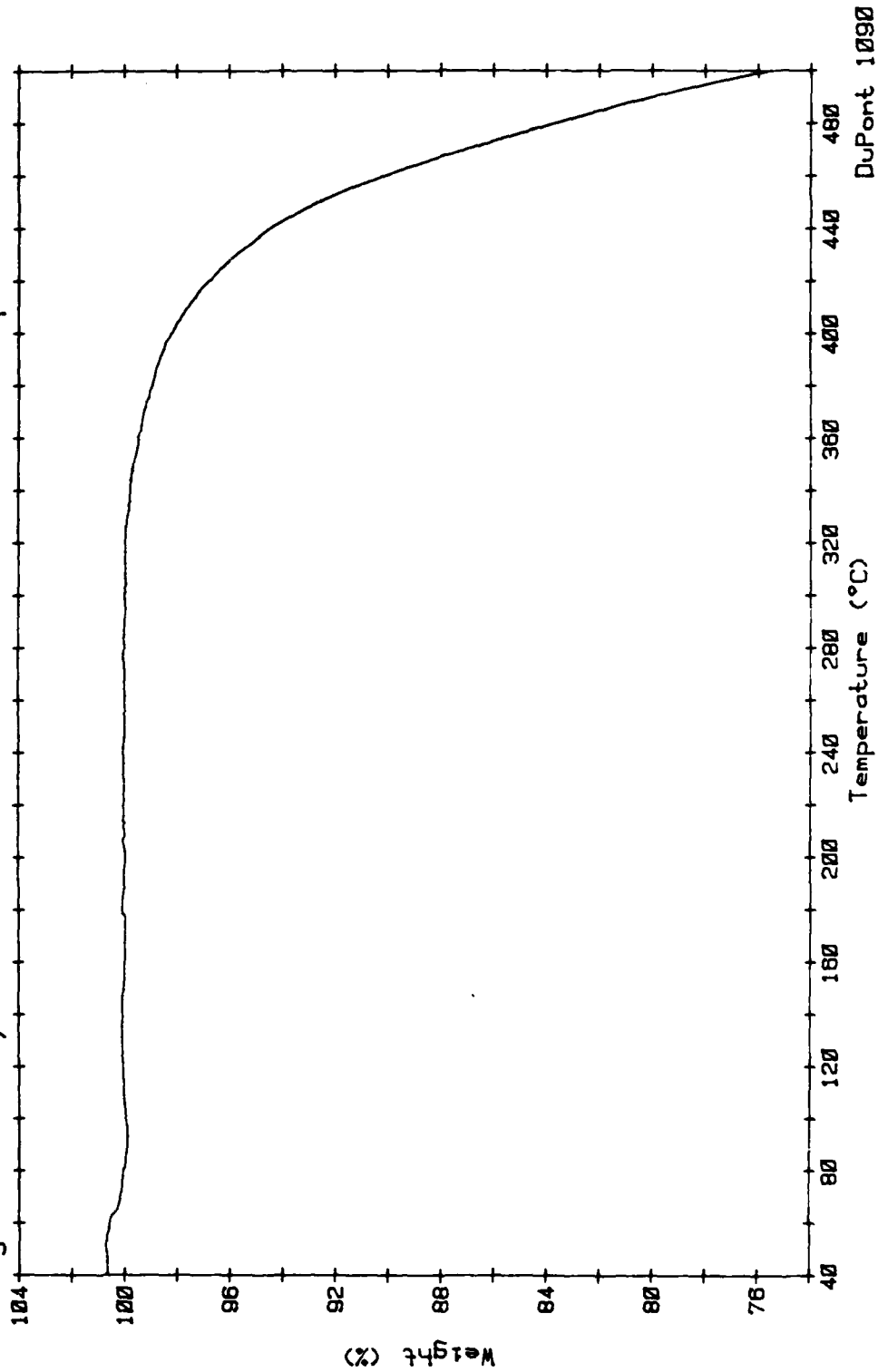


Figure 18. TGA in Air of AS4/T2000



Figure 19. AS4/T1000 After Ten Thermal Cycles (50x)



Figure 20. AS4/T2000 After Ten Thermal Cycles (50x)



Figure 21. SEM Fractograph of Tensile Failure (1000x)



Figure 22. AS4/T1000 Tensile Specimen

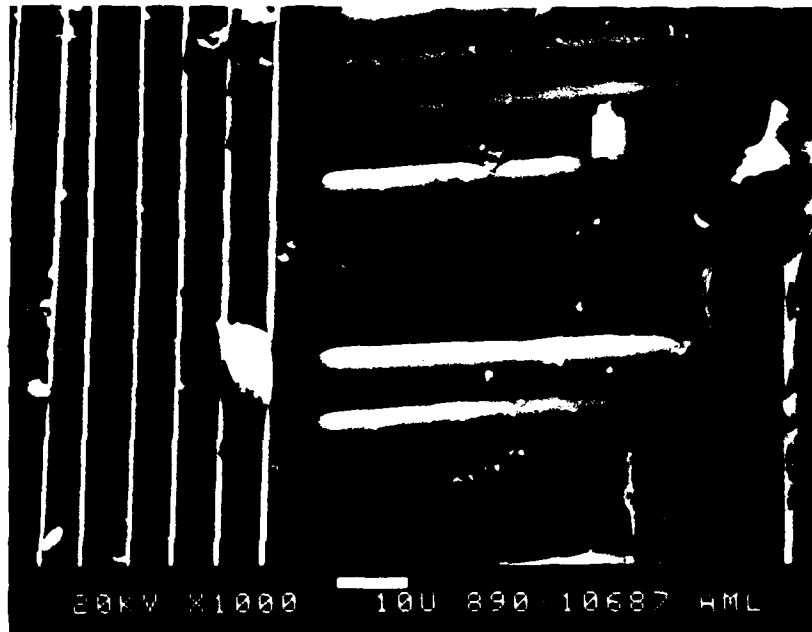


Figure 23. SEM Fractograph of AS4/T1000 DCB Specimen (1000x)



Figure 24. Microcracking in Conductive Resin Laminate (200x)

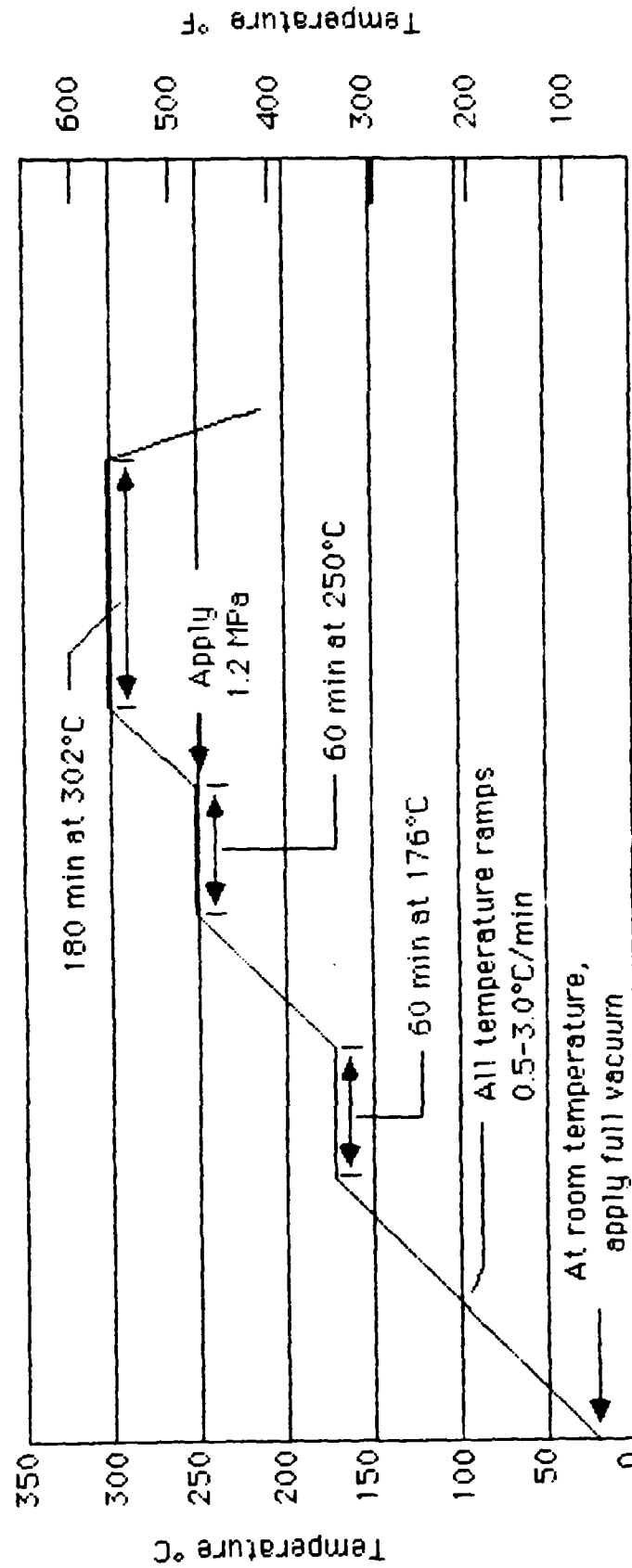


Figure 25. PMR-15 Autoclave Cure Cycle

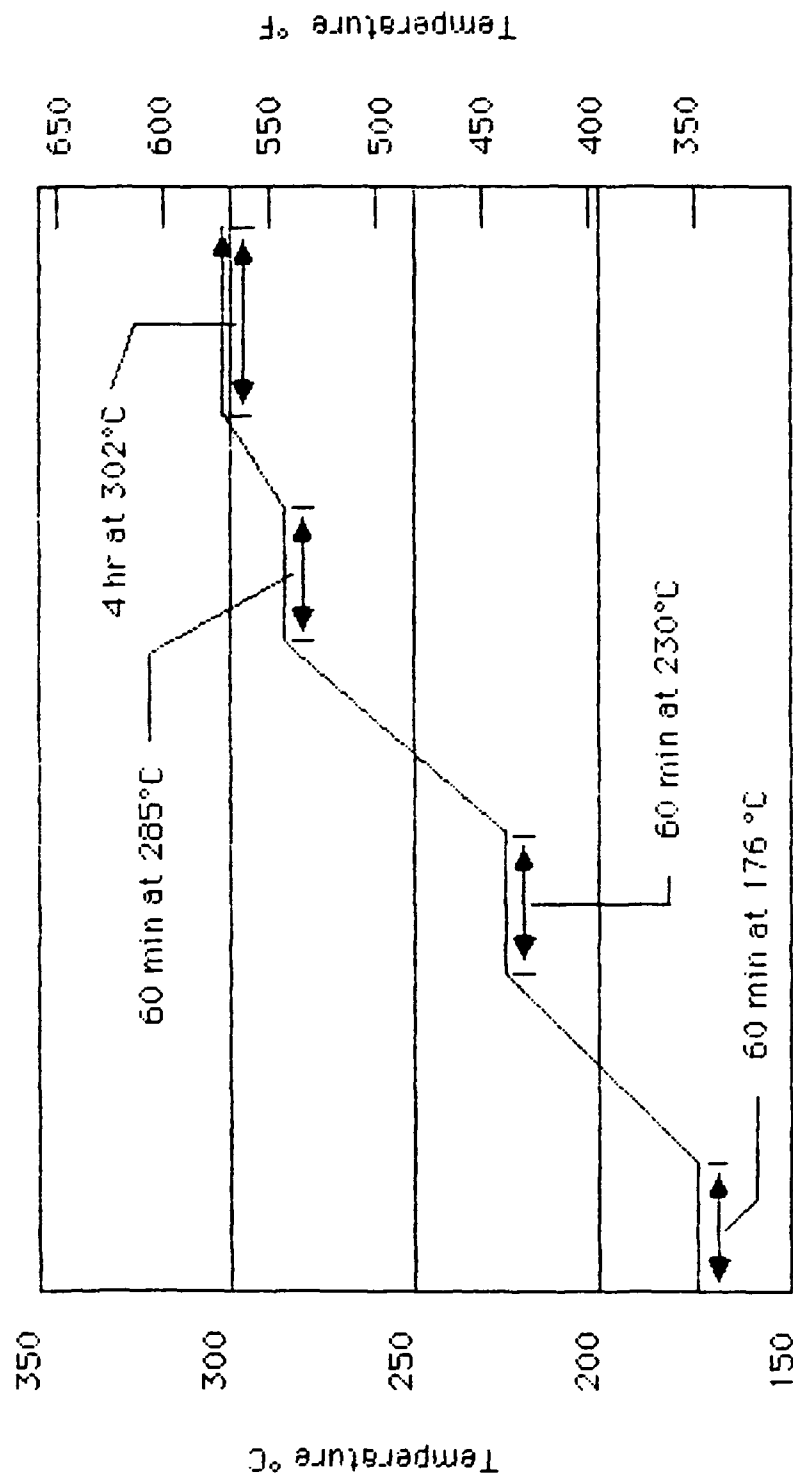


Figure 26. PMR-15 Postcure Cycle

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